Degree of Conversion of Luting Resins Around Ceramic Inlays in Natural Deep Cavities: A Micro-Raman Spectroscopy Analysis

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Clinical Relevance

Contrary to previous evidence, the degrees of conversion of resin luting materials have been shown to reach statistically similar values in three different depths around ceramic inlays luted to natural cavities. Both dual-cure and light-cure materials have presented conversion homogeneity, although they have been shown to be material-dependent.

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SUMMARY

This study evaluated the degree of conversion (%DC) of luting agents around ceramic inlays placed in deep natural cavities. Thirty-six cylindrical Class I cavities (diameter = 4 mm, depth = 4 mm) were prepared in freshly extracted human teeth and randomly divided according to the luting materials used for luting CAD/CAM fabricated inlays (Empress CAD). The dual-cure resin cements Clearfil Esthetic Cement and Variolink II Low and the light-cure composites Grandio Flow and Grandio were luted using the total-etch technique. The self-adhesive dual-cure cements RelyX Unicem and Maxcem Elite were used as recommended by the manufacturer. All of the restorations were photo-activated using a quartz halogen unit (Elipar TriLight; 750 mW/cm2) for 40 seconds. After 24 hour dry-storage in the dark, all the teeth were vertically sectioned into two halves (n=12 per group) using a slow-speed diamond-saw in the bucco-vestibular direction under constant water lubrication to

avoid specimen heating. The DC of the luting materials was measured by vibrational spectroscopy using a µ-Raman spectrometer at depths of 1, 3 and 4 mm on each side of the tooth halves (n=24). Disc-shaped samples were produced for measurement of the maximum %DC of each material. Two-way ANOVA and the Student-Newman-Keuls post-hoc test were used in the statistical analysis (α =0.05). All the materials showed no statistical differences in degree of conversion at all tested depths, except for Grandio Flow and Maxcem Elite. Dual-cure and light-cure luting materials showed polymerization homogeneity around ceramic inlays, although dual-cure conventional resin cements tended to show an overall higher conversion.

INTRODUCTION

When adhesively luting ceramic inlay/onlay restorations into deep cavities, many clinicians are uncertain whether the resin luting material has properly polymerized. Failure to polymerize the bonding and luting agents compromises retention and interface sealing, leading to clinical failure by means of restoration debonding and secondary caries.

Insufficient curing has been repeatedly associated with the poor mechanical properties of resin-based composites and is closely related to fatigue and wear resistance, ultimately affecting marginal integrity. Adhesive cements are also said to improve the integrity of the restorative space, allowing for a more effective stress transfer from the restoration into the supporting tooth. Consequently, poorly polymerized luting agents fail to establish a tooth-restoration unit, increasing the chance for restoration and cuspal fracture. Furthermore, less dense polymer networks result from inadequate polymerization, which can expedite the leaching of cytotoxic components that reach the pulp and lead to pulp inflammation.

Free-radical formation, which is responsible for the chain initiation step in polymerization reactions of light-activated luting resins, is dependent on the absorption of photons and the excitation of initiators (for example, camphorquinone), which, in turn, are dependent on the ability of light waves to reach the luting agent through the overlaying material. Light transmittance through ceramic restorations, especially in deep cavities, comes into focus, since light energy is decreased by absorption, reflection or scattering. Factors affecting light transmittance are contingent on practical aspects, such as ceramic thickness, selected shades and layering technique, as well as microstructure characteristics, namely crystalline structure, grain sizes, defects and intrinsic porosity. 10-12

Light transmission was found to be inversely related to ceramic thickness, 10-11,13 and percentages of less than 2% of transmitted light were reported under a 4 mm ceramic thickness.10 This dramatically reduces the available energy needed to convert initiators and start chain initiation reactions. Such influence of light attenuation through ceramic was also demonstrated via polymerization shrinkage rate curves of resin cements cured under ceramic discs of varying thicknesses.11 With increasing ceramic thickness, the peak time of polymerization shrinkage is significantly delayed and total shrinkage stress decreases.¹¹ This is a reflection of the inability of low energy delivery to convert the total amount of initiators within the resin matrix. Thus, the polymerization rate decreases as a function of initiator excitation, resulting in a higher percentage of uncured monomer in the matrix.

The extent of resin polymerization can be assessed by various methods, including mechanical microhardness testing¹⁴ or quantification of the degree of converted carbon double bonds.¹⁵ The degree of conversion (%DC) of a resinous material is thereby related to its mechanical properties^{13,16} and can be used as a parameter for predicting the clinical performance of adhesively luted restorations. FTIR (Fourier Transform Infrared Spectroscopy) and Raman vibrational spectroscopy are sensitive methods for providing detailed information of the transitions among the levels of vibrational energy of specimens. In the case of methacrylic resins, the energy transition of unreacted, remaining double bonds can be quantified by measuring peak changes in the vibration bands.¹⁷

Currently, the use of light-curing composites has been advocated as a technique for luting indirect restorations, since they allow a prolonged working time and better excess removal after insertion due to higher viscosity and improved color stability when compared to dual-cured cements.18 The major drawback of this technique relies on the lack of chemical initiators in light-curing composites—making their polymerization even more dependent on light energy transmitted through the restoration. It has been suggested by in vitro microhardness measurements that use of light-curing composites for luting indirect restorations could only be considered a safe method under optimal illuminating conditions:19 in other words, when luting thin, highly-translucent ceramic restorations with a high-power curing unit in close contact with the restoration surface.¹⁹ Otherwise, the use of a chemical catalyst is advised to increase monomer conversion.

Most of the studies evaluating the cure efficiency of resin cements through ceramic use metal molds¹⁹⁻²¹ and observe the measurements under increasing disc thicknesses. ^{12-13,22-24} This is a useful method for assess-

ing the degree of polymerization of a given luting agent at the bottom of a restoration, but its dissimilarity to the clinical situation fails to evaluate the quality of cure around it. The cement layer in the axial walls of a cavity is not covered by a uniform thickness of ceramic; therefore, its degree of polymerization may also be influenced by the transmission coefficient of the resin cement itself and the surrounding tooth structures during the light-activation step. Hence, the results from %DC measurements at the bottom of ceramic discs should not be extrapolated to interpretations of resin polymerization behavior under three-dimensional configurations as being a simplistic model. As a result, evaluating the degree of conversion of a luting agent in conditions that are closer to reality should be considered when clinically relevant results are desired.

The current study evaluated the degree of conversion of different classes of luting agents around ceramic inlays placed in deep natural cavities. The hypotheses tested were: 1) depth and location (axial or pulpal wall) have no influence on the degree of conversion and 2) light-cure and dual-cure luting agents yield a similar degree of conversion around ceramic inlays in deep cavities.

METHODS AND MATERIALS

Thirty-six freshly extracted caries-free human third molars were used in the current study. The teeth were debrided, examined to ensure the absence of defects and stored in antimicrobial 0.5% chloramine-T solution (Merck, Darmstadt, Germany) at 4°C for less than four weeks.

Cylindrical Class I cavities (diameter = 4 mm, 4 mm deep) were prepared using 80 µm-coarse diamond burs (Two-Striper Prep-Set, Premier, St Paul, MN, USA) under profuse water cooling and finished with a 25 µm-coarse finishing diamond bur. Rounded bottom angles were automatically prepared due to bur geometry.

For production of the ceramic restorations, the CAD/CAM technique was used (Cerec 3D, Sirona, Bensheim, Germany). The restorations were produced from Empress CAD presintered blocks (Ivoclar Vivadent, Schaan, Liechtenstein; shade LT A3), setting the luting gap to 150 µm.

After milling, all of the ceramic restorations were etched with hydrofluoric acid for 60 seconds prior to silanization (IPS Ceramic Etch & Monobond S, Ivoclar Vivadent). Both dentin and enamel were etched using 37% phosphoric acid (15 and 30 seconds, respectively), rinsed for 60 seconds and adhesively pretreated using the Syntac Classic system (Ivoclar Vivadent) according to the manufacturer's recommendations. The bonding agent was left uncured before the luting agent and restoration insertion. In the case of the self-etch luting resins, no adhesive was applied. Different classes of

lame/Manufacturer	Class	Application Mode	Filler Content/Size	Composition	Shade/Batch #
Clearfil Esthetic Cement, Kuraray	Dual-cured luting agent	Auto-mixture syringe	70% filler weight (49% vol)	Bis-GMA, TEGDMA, Methacrylated monomers, silanated glass fillers, colloidal silica, initiators, stabilizers	Universal/41111
Variolink II Low, Ivoclar Vivadent	Dual-cured luting agent	Manual mixture	73.4% filler weight (46.7% vol) 0.04-3.0 µm fillers with average size of 0.7 µm.	Bis-GMA, urethane dimethacrylate, triethylene glycol dimethacrylate, barium glass, ytterbium trifluoride, Ba-Al-fluorsilicate glass, Cemspheroid mixed oxide, catalysts, stabilizers	A3/L31516
Maxcem Elite, Kerr	Dual-cured self-adhesive luting agent	Auto-mixture syringe	69% filler weight	Methacrylate ester monomers, inert mineral fillers, ytterbium fluoride, activators and stabilizers	Yellow/3056148
RelyX Unicem, 3M ESPE	Dual-cured self-adhesive luting agent	Capsule	72% filler weight, average filler size <12.5 μm	Methacrylated phosphoric esters, dimethacrylates, acetate, initiators, stabilizers, glass fillers, silica, calcium hydroxide	A1/301337
Grandio Flow, Voco GmbH	Light-cured flowable resin composite	No mixture	80% filler weight (65.4% vol)	Bis-GMA, TEGDMA, HEDMA, fillers, initiators, stabilizers	A3/780798
Grandio, Voco GmbH	Light-cured hybrid resin composite	No mixture	87% filler weight (71.4% vol)	Bis-GMA, TEGDMA, HEDMA, fillers, initiators, stabilizers	A3/770810

resin luting materials (n=6) with different curing protocols (light-cured and dual-cured) were selected as luting agents (Table 1). The materials were applied to the cavity according to the manufacturers' recommendations. The ceramic restorations were inserted with finger pressure and immediately light-cured for 40 seconds occlusally at a distance of 1 mm from the restoration surface using a quartz halogen unit (Elipar TriLight, 3M ESPE, Seefeld, Germany; 750 mW/cm²; tip diameter = 9 mm) operating in standard mode. For insertion of the restorations luted with composite (Grandio, Voco GmbH, Cuxhaven, Germany), an ultrasonic insertion technique was used.¹⁸ Six teeth were randomly assigned to each luting resin and vertically sectioned into two halves (n=12 per group) using a slow-speed diamond-saw (Isomet, Buehler, Lake Bluff, IL, USA) in the bucco-vestibular direction under constant water lubrication to avoid specimen heating. The half-teeth were not polished to avoid post-curing development due to heating.

The degree of conversion at the selected regions was measured by u-Raman spectroscopy. Spectra were acquired from each half-tooth luting space following 24hour dry storage at 37°C in a dark environment. On each section, six measurements were recorded, three on each side (buccal and lingual/palatinal) at depths of 1 mm, 3 mm and 4 mm from the margins of the ceramic restorations (n=24). The 4 mm measurements were made at the pulpal floor 1 mm away from the cavity walls and 2 mm apart. Both the unpolymerized and polymerized resins were measured in backscattering geometry using a Micro-Raman spectrometer (Nicolet Almega XR Dispersive, Thermo Fisher Scientific Inc, Waltham, MA, USA) with a light microscope (Leica DMRXE, Leica Microsystems GmbH, Bensheim, Germany). Raman spectra were collected in the range of 1500-1800 cm⁻¹ using the 780 nm laser excitation line (Exposure time: 30 seconds: Accumulations: 4: Background exposures: 4; Aperture: 50 µm slit under a 100-fold microscope objective with NA 0.95).

To avoid interference with the spectra background, an automatic baseline correction was performed (Omnic software, Thermo Fisher Scientific Inc) for the calculation of the peak absorbance. The %DC was calculated according to the two-frequency technique using the net peak absorbance areas of the aliphatic C=C stretching vibrations at 1638 cm⁻¹ as analytical frequency and the aromatic $C\cdots C$ stretching vibrations at 1608 cm⁻¹ as reference frequency according to the equation:

%DC = 100 ·
$$\left(1 - \left(\frac{A_M (C...C) \cdot A_P (C = C)}{A_M (C = C) \cdot A_P (C...C)}\right)\right)$$
 (1)

where A_M and A_P represent the band height ratios of the uncured and cured material, respectively.

Ten discs (diameter = 10 mm, 2 mm thick) of each material were produced using a split metal-mold mounted on a glass slide and cured through a Mylar strip for 40 seconds. After 24 hour dry storage at 37° C, maximum %DC was determined using the above-mentioned method on the surface of each specimen.

Statistical analysis was performed using two-way ANOVA to assess interactions between the independent factors depth (1, 3 and 4 mm) and luting material on the %DC. The Student-Newman-Keuls multiple range test was used for multiple comparisons (α =0.05). Statistical analysis was calculated using SPSS 16.0 for Windows software (SPSS Inc, Chicago, IL, USA).

RESULTS

Table 2 shows the mean values and standard deviations of maximum %DC and %DC for both light- and dual-cured materials at the three depths tested.

Maximum %DC measured on the surface of disc samples showed no statistical difference in %DC at 1, 3 or 4 mm for all materials, apart from Grandio Flow (Voco GmbH) and Maxcem Elite (Kerr Corporation, Orange, CA, USA). For Grandio Flow, no difference was observed between maximum %DC and %DC at 1 and 2 mm, while maximum %DC of Maxcem Elite (Kerr Corporation) was significantly higher than the %DC measured at the three depth points of the interface.

Groups	Max DC%	Axial Walls		Pulpal Wall (4 mm)
		1 mm	3 mm	1
Clearfil Esthetic Cement	76.9 (1.9) a A	77.5 (8.3) a A	79.7 (6.2) a A	76.2 (10.2) a A
Variolink II Low	76.4 (1.9) a A	76.2 (2,2) a A	74.7 (3.0) a A	71.9 (4.0) a A
Grandio Flow	70.2 (2.3) a A	71.3 (3.4) a,b A	65.4 (4.6) b A,B	59.8 (3.9) b B
Grandio	63.6 (2.2) b,c A	65.7 (2.6) b,c A	64.5 (4.5) b A	58.6 (2.8) b A
RelyX Unicem	59.1 (3.0) c A	62.4 (2.1) c A	60.6 (4.1) b A	56.6 (1.4) b A
Maxcem Elite	61.1 (2.6) c A	44.3 (8.0) d B	35.1 (9.5) c C	29.8 (4.4) c C

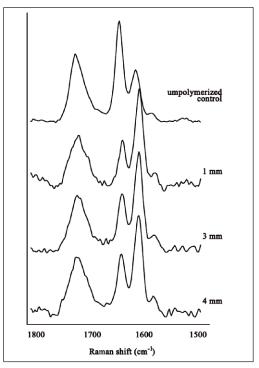


Figure 1. The Raman spectra of Variolink II unpolymerized and after polymerization at 1, 3 and 4 mm depths. Note the decrease in aliphatic peak vibration around 1638 cm⁻¹, characterizing the conversion of monomer into polymer chains. From 1 mm to 4 mm, a difference in the absorption peaks can be distinguished, although it is not statistically discernable.

Clearfil Esthetic Cement (Kuraray Co, Ltd, Tokyo, Japan) reached the highest values of conversion in all tested depths, while Maxcem Elite (Kerr Corporation) performed the worst. At a depth of 1 mm, no statistically significant differences could be detected for Clearfil Esthetic Cement, Variolink II and Grandio Flow. The two light-curing composites, Grandio and Grandio Flow (Voco GmbH), showed statistical similarity for 1, 3 or 4 mm tested depths. Maxcem Elite yielded the lowest values of conversion in all depths, as they were statistically different from all the other materials. RelyX Unicem performed worse than the dual-curing resin cements Clearfil Esthetic Cement and Variolink II (Ivoclar Vivadent) in all tested depths.

Within the same material, except for Grandio Flow at $1\ \mathrm{mm}$ and $4\ \mathrm{mm}$, all the materials showed no statistical differences for degree of conversion at the three measured depths.

In Figure 1, the Raman spectra of Variolink II demonstrates the decrease in aliphatic peak vibration to be around 1638 cm⁻¹, characterizing the conversion of monomer into polymer chains. From 1 mm to 4 mm, a difference in the absorption peaks can be distinguished, although they were not statistically discernable.

DISCUSSION

Due to light-activation being dependant on light transmittance and irradiation distance, 25-26 the degree of cure of luting agents around ceramic inlays in deep cavities is not expected to be homogeneous. The results of the current study seem to contradict this assumption, as either dual-cure or light-cure luting agents were able to achieve statistically similar degrees of conversion in the three tested location depths. This established assumption has, for years, been inaccurately built on studies of the depth of cure of bulk resin composites²⁷⁻²⁸ and the %DC of thick resin cement films cured under overlaying materials with increasing nesses. 12-13,22-24,29-30 Although valid for assessing the materials polymerization behavior of materials in controlled conditions, such experimental designs are not similar to the clinical scenario and, thus, are prone to misleading conclusions, as far as important variables are left aside, such as cavity configuration and light transmission through enamel/dentin during polymerization.

As light travels through the composite, light curing is progressively attenuated and depth of cure drops accordingly.^{26,31} Through ceramic restorations, light transmission has been shown to be attenuated from 91% to nearly 100% when ceramic discs from 1.5 to 4 mm of different shades were interposed between the light source and a photosensitive cell.10 Based on this logic, the values for degree of conversion at 3 mm and 4 mm should have been gradually lower when compared to 1 mm. Evoking previous studies on the polymerization degree of resin cements through ceramic discs, it would be natural to expect a drop in %DC with progressing depth. It was not without surprise, though, that, for the majority of the tested materials, no statistical differences were found for the three measured depths and the maximum %DC that led the authors of the current study to accept the first hypothesis.

The reasons behind how this supposed low energy delivery could result in such high conversion at 3 mm and 4 mm could only be explained by the methodology employed. Different from other studies, the authors of the current study measured the conversion rates in luting agents as thin films around restorations bonded to actual natural teeth. When it comes to clinical relevance, there is no question about the difference between measuring the degree of cure at the cavity walls of natural teeth and measuring the degree of cure of cements under ceramic discs in artificial molds. In the current study, light not only went through the ceramic, but also through enamel, dentin and the respective composite. All of these substrates have different light absorption coefficients, which would influence the energy reaching different areas. Also, light leaving the curing tip peripherally to the 4 mm diameter of the ceramic inlay also penetrated dental tissues,

and by internal scattering, may have contributed to increased energy delivery to the luting agent around the restoration.

It is not yet fully understood how polymerization reaction kinectics and chain reaction propagation of resin composites in thin films occur under such constrained geometries. As in diffusion-controlled networks, the reaction rate is determined by the rate of transport of the reactants through the medium; changes in geometry of the medium may affect the reaction direction. In thin, constrained resin films, the available reactants (monomers) are available in a higher concentration in a two-dimensional plane (laterally and axially) as compared to a three-dimensional plane found in bulk materials. In this way, polymer chain growth may be predisposed to propagate towards the depth of the cavity, thus increasing conversion rates counteracting for the reduced concentration of growth centers due to poor initiator excitation. Li and others,32 using digital image correlation, have shown that the polymerization rate of unconstrained bulk resin composites may occur nonuniformly at constant depths. Differences in transverse and axial linear shrinkage strains were detected even when minimal friction was assured, causing restrain in the axial direction to influence transversal shrinkage.³² This evidence, in conjunction with that found in the current study, supplies primary indications, other than only energy/initiator dynamics, that other factors may be involved in the polymerization reaction within resinfilled materials. If unconstrained shrinkage can influence the direction of polymerization chain propagation, it is reasonable to speculate the possible heterogeneity arising in highly constrained high C-factor configurations. Therefore, further efforts must be focused on clarifying this issue.

In thin films around thick ceramic restorations, deeper areas should polymerize at lower rates compared to areas closer to the light source and, as a result, stress development is supposedly not uniform. Although high conversion degrees would be desired for good material properties buildup, higher %DC are related to higher shrinkage stress, 33-34 imposing the adhesive layer to a greater challenge.

The %DC of some materials under investigation seem to be higher than those found in the aforementioned studies, which measured the %DC of resin cements cured through ceramic discs as thick as 4 mm. Furthermore, the maximum values of %DC for dimethacrylate-based dental composites have been found not to surpass 50% when measured with FTIR and Raman spectroscopy techniques. Many studies, however, have found degrees of conversion for dental composites reaching values higher than 60% in optimal conditions. Spinell and others, using an FTIR spectrometer, also reported %DC values ranging from 49.6% to 73.2% of resin cements in self-cure mode and from

67.3% to 85.1% in dual-cure mode.³⁹ In this sense, the degree of conversion has been shown to be highly material-dependent,³⁷ as differences in filler size and fraction/percentage,⁴⁰ photoinitiation chemistry,³⁸ resin formulation and organic monomer type^{17,41} play a fundamental role in the polymerization process.

Other factors, such as ceramic microstructure, shade, and translucency, may also influence the %DC of resin composite under ceramic restorations. 10,12,19 The effect of such variables has been shown to be clinically relevant, but little impact on %DC homogeneity depth-wise should be expected. 12 In this way, although only one type and shade of ceramic has been employed in the current study, it can be speculated that the results from this study could be extrapolated to other types of ceramic materials.

In order to prevent further heating influence on %DC during sectioning, copious water irrigation was used, with a diamond saw rotating at low speed. Even so, the sectioning influence could not be ruled out as a contributing factor for the conversion values found herein, although its influence seems to have been minimal, given, for instance, the low values found for Maxcem Elite and the differences observed at the respective depths for the same cement. One hypothesis for the high values of the degree of conversion found for some materials in the current study is related to the interaction between the adhesive and the luting agents during polymerization. The unpolymerized adhesive layer coating the cavity walls most probably diffused into the luting agent during placement of the restoration, providing the restorations with a greater supply of initiators, increasing the number of polymerization growth centers, thus resulting in a higher %DC. Furthermore, the effects of the adhesive on %DC might have been more pronounced at the pulpal floor, since %DC values near the surface were closer to the maximum %DC. This may also explain the lower %DC reached by the self-adhesive cements, as compared to the luting agents used in conjunction with the adhesive agent. However, the real influence of the adhesive on the cement conversion must be further clarified.

Regarding %DC homogeneity, the two material exceptions were the light-curing composite Grandio Flow and the self-adhesive cement Maxcem Elite. When compared to other materials, Maxcem Elite presented statistically lower degrees of conversion in all tested depths—all beneath 55%. Its conversion values were lower than light-cured composites, which may reflect problems in its photoinitiator system. Grandio Flow showed statistical differences between 1 mm and 4 mm, but all values were beyond 55%.

The values for degree of conversion in luting agents with different polymerization modes (light-cured and dual-cured), which were observed in the current study

using µ-Raman spectroscopy, also led the authors to accept the second hypothesis. Although Grandio Flow and Grandio yielded statistically lower conversion values than Clearfil Esthetic Cement and Variolink Low at 3 mm and 4 mm, at the same depth, they were comparable to the dual-cured cement RelyX Unicem and statistically higher than Maxcem Elite. However, as values of %DC have been shown to be material-dependent, it may not be prudent to state that light-curing composites can reach the same curing efficacy as dual-cured composites, even in natural cavities. RelyX Unicem, for instance, has been shown to reach a lower %DC than other dual-cure cements when directly light-activated, and it has been shown to have compromised chemical curing. ^{39,42}

Many conversion studies have shown the beneficial role of incorporating chemical activator in luting agents, 12,19 especially in non-optimal conditions. In dualcure cements, chemical cure accounts for the remaining free radicals and double bonds created by irradiation added to the continued reaction precipitated by chemical activation. After a 24-hour period, chemical cure in dual-cure materials could be expected to be more efficient in continuing the chain propagation process to a higher %DC. This can be clearly distinguished in the current study by comparing the light-curing materials with Clearfil Esthetic Cement and Variolink II Low at 3-mm and 4-mm depths. Therefore, although the lightcuring composites tested in the current study could reach acceptable degrees of conversion around ceramic inlays up to 4 mm in depth, their indication for luting indirect restorations should still be exercised with caution. Longitudinal results of a clinical investigation have shown that the performance of ceramic inlays luted with a light-curing composite was comparable to inlays luted with dual-cure resin cements after eight years of observation.43 Four years later, a significant difference was detected between luting techniques in which a significantly higher number of inlays luted with light-curing composite underwent catastrophic bulk fractures. 44 This can be a sign of the long-term beneficial effect of continued chemical cure present in dualcure resin cements for improving fatigue resistance at the interface.

CONCLUSIONS

Within the limitations of the current study, it is possible to conclude that some resin-based luting agents can reach %DC homogeneity, with progressing depth around ceramic inlays in deep natural cavities. As with dual-curing materials, light-curing composites can be used to lute ceramic inlays up to 4-mm deep.

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